- Draft Standard Study Plan -

TEST ITEM NAME

Anaerobic Transformation in Soil

acc. to OECD 307 Guideline for Testing of Chemicals
(adopted: 24th April 2002)

Sponsor

TELOMER RESEARCH PROGRAM
C/O RAND CORPORATION
1200 South Hayes Street
Arlington, Virginia 22202
USA

Test Facility

DR.U.NOACK-LABORATORIEN
Käthe-Paulus-Straße 1
D-31157 Sarstedt

Laboratory Project ID

Project-No. XXXXXXX
Study-No. ASBXXXX-

Date

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1 Test Facility Information and Study Sponsor

TEST FACILITY	DR.U.NOACK-LABORATORIEN Käthe-Paulus-Str. 1 D-31157 Sarstedt
Study director	Silke Fiebig (Engineer, Biotechnologist) Address see above
Scientists	Dirk Schulze (Chemical Engineer) responsible for LC-MS/MS analysis Address see above Thomas Geffke (Chemical Engineer) responsible for GC-MS analysis Address see above
Test facility management	Dr. Udo Noack (Biologist) Address see above
Head of quality assurance	Gudrun Möhrmann-Kalabokidis (Biologist) Address see above

SPONSOR	TELOMER RESEARCH PROGRAM (TRP) C/O RAND CORPORATION 1200 South Hayes Street Arlington Virginia 22202 USA
Coordinator	Dr. Kayo Kusumi DAIKIN INDUSTRIES, LTD
Monitor	Dr. Seiji Shin-ya for ASAHI GLASS CO., LTD. Dr. Volker Koch for CLARIANT GMBH Dr. Kayo Kusumi for DAIKIN INDUSTRIES Dr. William R. Berti for DUPONT CHEMICAL SOLUTIONS ENTERPRISE

2 Test Item Characterisation

Test items will be telomer based polymers and characterized acc. to 2.1 in a CBI study plan supplement.

2.1 Characterisation Data of Test Item

Test Item

Source of Test Item

Batch Number

CAS Name

CAS RN

Material Number

Chemical Purity

Chemical Characterisation

Stability

Appearance / Colour

Residuals / Potential Transformation Products see section 2.2

Total Fluorine Content (Polymer only)

Total Fluorine Content (Dispersion)

Total Carbon Content (Polymer only)

Total Carbon Content (Dispersion incl. additives)

Average Molecular Weight

Molecular Weight Distribution of Polymer

Certificate of Analysis Date

Expiry Date

Date Received

Recommended Storage

Storage at Testing Facility

Identification Parameter

The test item and the information concerning the test item were provided by the sponsor.

2.2 Characterisation Data of Residuals and Potential Transformation Products

The detailed chemical characterisation of residuals in the test item as well as potential transformation products being formed during the test are given in tables 1-4.

Table 1: **Potential Residuals and Transformation Products**

Short Cut	CAS Name	Content in Test Item	
		in µg/g	in nmol/g
8-2 OH	1-Decanol, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro- (7CI, 8CI, 9CI)	will be specified in a study plan supplement	
8-2 Acrylate	2-Propenoic acid, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluorodecyl ester (9CI)		
8-2 Iodide	Decane, 1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-10-iodo- (8CI, 9CI)		
8-2 Olefine	1-Decene, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro- (8CI, 9CI)		
8 Iodide	Octane, 1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-8-iodo- (9CI)		
8 COOH	Octanoic acid, pentadecafluoro- (8CI, 9CI)		

Table 2: **Potential Transformation Products**

Short Cut	CAS Name	Content in Test Item	
		in µg/g	in nmol/g
8-2 COOH	Decanoic acid, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro- (8CI, 9CI)	will be specified in a study plan supplement	
8-2 U COOH	2-Decenoic acid, 3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-hexadecafluoro- (9CI)		
7-3 COOH	CF3(CF2)6CH2CH2COOH	Not expected to be present in test item. Analytical standards are not currently available.	
7-2 sOH	CF3(CF2)6CHOHCH3		

3 Analytes

The potential biotransformation of the telomer based polymers will be determined by monitoring the below specified analytes. The short cuts given for the analytes will be used for simplification.

3.1 Perfluoroctanoic acid

Abbreviation	PFOA
Short Cut	**8 COOH**
CAS RN	335-67-1
Source	SIGMA ALDRICH
Molecular formula	$C_8HF_{15}O_2$
Molecular weight	414.07g/mol
Water solubility	3.4 g/L (3M report)
pKa	2.5 (3M report)
pH-value in water	2.6 (3M report)
Vapor pressure	10 mm Hg at 25 °C (3M report)

3.2 2H,2H-Perfluorodecanoic acid

Abbreviation	2-PFOEA
Short Cut	**8-2 COOH**
CAS RN	27854-31-5
Source	CLARIANT GMBH, Werk Gendorf
Molecular formula	$C_{10}H_3F_{17}O_2$
Molecular weight	478.10 g/mol

3.3 2H-Perfluoro-2-decenoic acid

Abbreviation	2H-HDF-2-DA
Short Cut	**8-2 U COOH**
CAS RN	70887-84-2
Source	CLARIANT GMBH, Werk Gendorf
Molecular formula	$C_{10}H_2F_{16}O_2$
Molecular weight	458.10 g/mol

3.4 2-Perfluorooctylethanol

Abbreviation	C8-2 TB Alcohol
Short cut	**8-2 OH**
CAS RN	678-39-7
Source	CLARIANT GMBH, Werk Gendorf
Molecular formula	$C_{10}H_5F_{17}O$
Molecular weight	464.11 g/mol

3.5 8-2 Alcohol Acrylate (if applicable)

Abbreviation	C8-2 TB Acrylate
Short cut	**8-2 acrylate**
CAS RN	27905-45-9
Source	CLARIANT GMBH, Werk Gendorf
Molecular formula	$C_{13}H_7F_{17}O_2$
Molecular weight	518.16 g/mol

3.6 7-2 Secondary Alcohol

Abbreviation	Secondary C7-2 TB Alcohol
Short cut	**7-2 sOH**
CAS RN	24015-83-6
Source	not available
Molecular formula	$C_9H_5F_{15}O$
Molecular weight	414.10 g/mol

3.7 7-3 Acid

Abbreviation	7-3 Acid
Short Cut	**7-3 COOH**
CAS RN	812-70-4
Source	not available
Molecular formula	$C_{10}H_5F_{15}O_2$
Molecular weight	442.11 g/mol

4 Objectives of the Study

TITLE	Test Item: Anaerobic Transformation in Soil
GUIDELINE	The study will be based on the following guideline: OECD 307 Aerobic and Anaerobic Transformation in Soil (24th April 2002), but will be adapted according the specific needs related to the test item. The recommendations given by the guideline will be followed as closely as possible.
TYPE AND PURPOSE OF THE STUDY	The purpose of the study is to determine the biotransformation potential of the telomer-based polymers in soil under anaerobic conditions and controlled laboratory conditions over a period of 12 months. The potential biotransformation of the test item will be determined by monitoring the analytes 8 COOH, 8-2 COOH and 8-2 U COOH via LC-MS/MS analysis and C8-2 OH by GC-MS analysis. The 8-2 acrylate will be monitored for test items in which it is constituent or a residual.

5 Test System

DESCRIPTION OF TEST SYSTEM	Freshly collected soil from the top 20 cm layer with an organic carbon content of > 2.0 % and a microbial biomass of at least 1 % of total organic carbon or other appropriate soil, e.g. Mollisol from USA. The soil will be characterized for texture, pH-value, cation exchange capacity, organic carbon, bulk density, water retention characteristics and microbial biomass.
Reason for the choice of the test system	The soil has a high microbial activity and will meet the requirements of the test guidelines.
Soil handling	The soil will be manually cleared of large objects and then sieved to a particle size of 2 mm (unless already done by the soil deliverer). The soil moisture content, the maximum water holding capacity (acc. SCHINNER (1993)) and the pH-value will be determined. Dry out of the soil is prevented by moistening with water once per week until test start if necessary. The soil will be checked for a detectable microbial biomass (result in terms of percentage of total organic carbon).

To re-establish the equilibrium of the microbial metabolism the soil will be preincubated at test temperature for 2 - 28 d before initiation of the definitive study. The duration of the preincubation period will be determined by measurement of glucose induced respiration rate. If storage is necessary the soil will be stored in the dark at 4 ± 2 °C for a maximum of three months. However before test start the soil will be adjusted to test temperature for at least 2 days.
Storage and pre-incubation time together will not exceed 3 months.

6 Test Groups

TEST ITEM	Test Item The test item as dry polymer with a nominal mean particle size of less than 500 µm will be used for the study.
Test concentration	The highest concentration tested in a respiration inhibition study shown to be non-inhibitory, or no greater than 10000 mg/kg soil dry weight, whichever is greater. The exact concentration will be determined in consultation with the respective study monitor of the company providing the test item. The definite test concentration will be specified in the study plan supplement.
CONTROL	Untreated soil (for specific analysis of the 5 analytes)
ABIOTIC TEST ITEM CONTROL	Sterilized soil (irradiated with ^{60}Co and dosing of sterilizing agent) treated with test item.
STERILE SPIKE RECOVERY CONTROL	Sterilized soil (irradiated with ^{60}Co and dosing of sterilizing agent) without test item and treated with 8-2 OH, 8-2 Acrylate (if applicable), 8 COOH, 8-2 COOH, 8-2 U COOH at 10 x LOQ. The sterility of the soil will be checked by determination of the mineralization and colony forming units (CFU) at test start and test end. Details of the soil sterilization procedure will be documented in the raw data and given in the report.
POSITIVE CONTROL	Untreated soil to check the activity of the microbial biomass at each sampling time.

7 Method

Incubation system/ vessels	Incubation system will be a static system. Test vessels will be Test PTFE-free glass serum bottles with foil lined gas-tight septum caps. Separate vessels will be used for each replicate and each sampling time. The complete content of the vessels will be extracted for analysis. Details of the incubation vessels (e.g. volume) will be documented in the raw data and given in the report.
Further laboratory equipment	Polypropylene laboratory equipment will be used where possible. To avoid cross contamination disposable labware will be used if available. Non-disposable labware will be cleaned acc. to a specific cleaning procedure (SOP CTRPGG).
Identification	Each test unit will be uniquely identified with at least study number, treatment and replicate number.
Replicates	Duplicates per sampling time
Amount of soil per incubation flask	ca. 20 - 30 g The definite amount depends on the sampling for the specific analysis, it will be documented in the raw data and given in the report.
Test duration	12 months
Temperature	20 ± 2 °C in the dark, in a temperature controlled laboratory room.
Soil moisture content	At the beginning of the test the soils will be adjusted to 40 - 60 % of the maximum water holding capacity. All incubation flasks will be checked in appropriate intervals (at least monthly) for losses by evaporation. Replicates will be weighed for this procedure. Sterile-filtered demineralised water will be added as necessary to compensate for water losses. If compensation is necessary, the headspace will be sampled before the vessels were opened.
Maintenance of anaerobic conditions	The incubation system will be purged with anaerobic gas (mixture of nitrogen and hydrogen) at test start. To check the anaerobic conditions the pH, oxygen concentration and redox potential (if possible) of the controls will be measured at each sampling time. To avoid O_2 leakage into the test vessels, the vessels will be stored in an anaerobic chamber.

Application

The test item or analytes of interest will be applied to the soil within test vessels once at test start.
The test item as dry polymer with a nominal mean particle size of less than 500 µm will be administered by direct weight addition.
The test item treatments will be prepared by dosing a weight of soil with sufficient test item to deliver the desired amount of test item.
The test vessels will be sealed with aluminium foil-lined septum, mixed and incubated.
The soil moisture content will be checked and adjusted, if necessary, prior to the addition of the test item or analytes of interest. Degassed sterile-filtered demineralized water will be added as necessary to compensate for water.
As far as possible the anaerobic treatments and controls will be prepared within an anaerobic environment.

The spike recovery controls will be prepared by dosing a weight of sterilized soil with appropriate volumes of 8-2 OH and 8-2 acrylate (if applicable) stock solution and 8 COOH, 8-2 COOH, 8-2 U COOH stock solution. The stock solutions will be injected directly into the soil using a glass microsyringe to the individual test vessels. The test vessels will be immediately sealed with aluminium foil-lined septum and the content of the vessels will be mixed.

Further details of the application technique and exact amounts of the test item and analytes of interest to be added will be documented in the raw data and given in the report.

7.1 Performance of the Study

COURSE OF THE STUDY

The soils will be sieved after collection and prior to application of the test item and analytes of interest. Moisture content, maximal water capacity, pH-value and CO_2-evolution of the microbial biomass will be determined prior to test start.

At test start the test item will be applied to the soil (see application). Untreated soil samples will be incubated under the same conditions (anaerobic) as the treated soil samples.
Duplicate incubation flasks will be removed at appropriate time intervals and analysed for the analytes of interest. The entire vessel incl. the foil-lined septum will be extracted.

To remove volatilised organic compounds, an exact volume of the headspace of the incubation vessels will be filtered through C18 Alltech® Maxi-Clean™ cartridges to trap potential volatile transformation products. At each sampling time prior to opening the vessel, the septum will be pierced with a needle connected to a C18 cartridge and a syringe. An exact volume will be filtered through the cartridge and the cartridge subsequently extracted and analysed. Further details of the method will be documented in the raw data and given in the report.

Losses by evaporation will be compensated by weighing the incubation flasks in appropriate intervals and moistening the soils with Sterile-filtered oxygen-free demineralised water if necessary. If moistening is necessary, the headspace will be sampled before the vessels were opened.
The anaerobic conditions in the headspace will be checked on regular time intervals (at least monthly) by measuring the residual oxygen content and CH_4 production. Further details of the method will be documented in the raw data and given in the report.

To check the activity of the biomass the CH_4 -evolution of the positive control will be carried out directly after application and at each sampling point (see below).
Biotransformation will monitored by specific analysis of the analytes of interest at sampling times as specified below.

Incubation will take place in a temperature range between
20 ± 2 °C in the dark.

TECHNICAL EQUIPMENT (Biological part)

pH-Meter, WTW Multi 350i
Thermohygrograph, LUFFT
Room air conditioner split type, FUJITSU
Other appropriate models are possible.

7.2 Type and Frequency of Measurements

TYPE OF DETERMINATIONS

The incubation temperature will be documented continuously with a thermo-hygrograph.

Soil moisture content will be checked periodically by weighing of the incubation flasks and adjusting with sterile-filtered oxygen-free demineralised water if necessary.

To check the biomass activity glucose induced respiration rates or other appropriate parameter (e.g. methane evolution) of the positive control will be determined. Details of the method will be documented in the raw data and given in the report.

Specific LC-MS/MS analysis of 8 COOH, 8-2 COOH, 8-2 U COOH and 7-3 acid will be performed on the test item, control, sterile spike recovery control and sterile test item control replicates. The entire test vessel incl. the foil-lined septum will be extracted.

Specific GC-MS analysis of 8-2 OH, 8-2 acrylate (if applicable) and 7-2 sOH will be performed on the test item, control, sterile spike recovery control and sterile test item control replicates. The entire test vessel incl. the foil-lined septum will be extracted.

The headspace will be filtered through C18 Alltech® Maxi-Clean™ cartridges. 8 COOH, 8-2 COOH, 8-2 U COOH, 8-2 OH, 8-2 acrylate (if applicable), 7-3 acid and 7-2 sOH will be determined by specific LC-MS/MS and GC-MS analysis, respectively.

SAMPLING SCHEDULE

Samples of soil and headspace gas for specific analysis of the analytes of interest will be taken on day 0, 7 and 14 and after 1, 2, 4, 6, 9 and 12 months (= 9 sampling times). The entire incubation vessels will be removed for analysis.

MEASUREMENT OF GLUCOSE INDUCED RESPIRATION RATES

Soil samples of each replicate will be mixed with a sufficient amount of glucose (range 2000 - 4000 mg/kg, concentration will be given in the report) to produce an immediate maximum respiratory response. 200 g soil will be filled into 500 mL glass flasks and closed with OXITOP® sensors. CO_2 will be adsorbed by sodium carbonate deposited in the headspace. Due to the adsorption of CO_2 and the oxygen uptake by the soil the pressure in the glass flasks will be reduced and measured. Based on the change of pressure the evolved CO_2 and thus the consumed O_2 will be calculated. Incubation will take place for 24 h in the dark at 20 ± 2 °C. The pressure will be measured 360 times in 24 hours after glucose supplement. Further details of the method will be documented in the raw data and given in the report.

8 Specific Analysis

DEMONSTRATION OF ANALYTICAL CAPABILITY

The recovery of 8 COOH, 8-2 COOH, 8-2 U COOH from the extracts will be demonstrated in the sterilized test system. Each analyte will be added at a concentration not to exceed 10-fold the LOQ. After a minimum of 24 h of mixing the recovery should be 70 - 130 % (in compliance with the US EPA Quality System for Environmental Data and Technology).

VALIDATION

All analytical methods will be validated prior to the test acc. to SOP CTRPMV and relevant guidelines e.g. EPA quality guidelines and SANCO/3030/99. Details of the validation will be documented in the raw data and given in the report.

SPIKE CONTROLS

Controls freshly spiked at LOQ level will be analysed with each set of data (middle of data set).

DILUTION OF SAMPLES

If sample concentrations are outside the calibration curve range, the samples will be diluted and re-analysed.

8.1 LC-MS/MS Analysis

Extraction method	Will be specified in a study plan amendment.		
Equipment	HPLC	:	2695 Separations Module, WATERS
	Detector	:	Mass selective detector, Micromass Quattro Premier™ (MS/MS-detector), WATERS
	Software	:	MassLynx™ 4.0
Reagents	HPLC water Methanol Acetic acid		
Analytical column	C18 reversed phase column, 2.1 mm x 40 mm, 3 µm		
Mobile phase	A = Acetic acid 0.15 (v/v) B = Acetonitrile		
Injection volume	5 µL		

Further details of the complete analytical method will be documented in the raw data and given in the report.

8.2 GC-MS Analysis

Extraction method	Will be specified in a study plan amendment.	
Equipment	Gas chromatograph	: CP-3800, VARIAN
	Autosampler	: CTC Combi PAL with SPME option, CTC ANALYTICS
	Detector	: MS, Saturn 2000, VARIAN
	Software	: Saturn GC/MS Workstation 5.52, VARIAN
Reagents	1-methyl-2-pyrrolidone (NMP)	
Analytical Column	Factor Four Capillary Column (VARIAN), 30 m, i.d.: 0.25 mm,	
Column	VF-35ms (VARIAN), 30 m, i.d.: 0.25 mm, Film thickness: 0.25 µm	
Injector	Splitless for 1.5 min (10.0 psi pressure pulse for 1.5 min)	
Injector temperature	250 °C	

Oven programme

Temperature [°C]	Heating rate [°C/min]	Hold time [min]
40	0	1.5
90	10	0.5
200	50	1.0

Carrier gas Helium 1 mL/min column flow

Run time 10.2 min

Retention time Approx. 7.4 min

Further details of the complete analytical method will be documented in the raw data and given in the report.

8.3 Analysis of Headspace Gas

Extraction method Alltech® Maxi-Clean™ cartridges will be extracted and the extract will be analysed for the analytes of interest.

Further details of the complete analytical method will be documented in the raw data and given in the report.

8.4 Determination of Total Organic Fluorine

Total organic fluorine will be calculated from the difference of total fluorine and fluoride.

TOTAL FLUORINE ANALYSIS

Total fluorine analysis acc. to WICKBOLD TORCH will be carried out from a well mixed sample which is decomposed or volatilised up to 2000 °C in the presence of wet oxygen and swept through an oxyhydrogen flame. Combustion products are collected and fluoride will be determined with an ion selective electrode.
The total fluorine analysis will be carried out at CLARIANT GMBH, Werk Gendorf at NON-GLP-State. Details of sample preparation and analytical method will be given in an annex to the report.

FLUORIDE ANALYSIS	Inorganic fluoride will be determined with a fluoride selective electrode. Details of the sample preparation and the analytical method will be documented in the raw data and given in the report.

9 Time Schedule

STUDY INITIATION DATE	Will be specified in a study plan supplement.
PROPOSED EXPERIMENTAL START DATE	Will be specified in a study plan supplement.
PROPOSED EXPERIMENTAL COMPLETION DATE	Will be specified in a study plan supplement.
PROPOSED DRAFT REPORT DATE	Will be specified in a study plan supplement.

10 Study Plan, Changes

STUDY PLAN SUPPLEMENT	Test item specific information will be given in a study plan supplement, which will be signed by the study director, scientists and the appropriate study monitor.
DEVIATIONS OF THE STUDY PLAN	Unplanned changes of the study plan will be identified in writing, signed by the Study Director and communicated to Study Monitor as soon as possible. Any deviation statement will include the reason for the deviation, its date of occurrence and its anticipated effect on the outcome of the study. The deviations will be included in the study records. The chapter "Deviations of the Study Plan" in the final report will reflect every deviation, the reason for the deviation and its anticipated effect on the outcome of the study.

AMENDMENT PROCEDURE

All amendments to this study plan will be described in detail by the Study Director prior to implementation and will contain the following information:

- a description of the study plan amendment
- the reasons for the study plan amendment
- impact of the changes on the study
- the signature of the Study Director and the effective date
- the Study Monitor must be notified of the study plan amendment

11 GLP and Reporting

SOP

The test facility will be responsible for Standard Operating Procedures (SOPs) during the study. Standard Operating Procedures will be in place for all phases and activities performed at DR.U.NOACK-LABORATORIEN.

QAU

Quality assurance of the study will be the responsibility of DR. U.NOACK-LABORATORIEN and will be carried out in compliance with the present OECD, EC and German principles of Good Laboratory Practice and DR.U.NOACK-LABORATORIEN standard operating procedures. The Quality Assurance Unit (QAU) of DR.U.NOACK-LABORATORIEN must provide written reports of all inspections to the Study Director. Phases to be inspected may include but are not limited to exposure of the test system to the test item, data collection, and reporting. Any problems found during the course of an inspection which are likely to affect study integrity shall be immediately brought to the attention of the Study Director and DR.U.NOACK-LABORATORIEN management. The Study Director will be responsible for reporting findings affecting study integrity to the Study Monitor as soon as possible. A QAU-statement will be included in the report.

GLP STATEMENT

The study, except for total fluoride analysis, will be conducted and reported in compliance with the present OECD, EC and German principles of Good Laboratory Practice which are consistent with US Good Laboratory Practice Standards and Japan Ministry of Economy, Trade and Industry. Signatures of the study director and scientists will attest to the authenticity of the study.

REPORTING

A Confidential Business Information (CBI) version and a non-CBI version of the report will be issued for each test item.
Generally the final report will include but not be limited to the following:

- study title
- name and address of the test facility
- name and address of the sponsor
- study initiation and completion dates
- start and end dates for the experimental part of this study
- test guideline(s) followed in conducting the study
- name of the study director, scientists, study monitor, and other personnel involved in the study
- objectives and procedures stated in the study plan, including amendments and major deviations from the study plan
- complete identification of test item identified as specified under 2.1, and a copy of the Certificate of Analysis for the test item, as provided by the Sponsor
- description of the test system
- description of the experimental design and all procedures used during the conduct of the study, including test item formulation, dispersion, and application information, test vessels, environmental parameter monitoring, data collection, sample collection
- description of testing conditions, including temperatures
- description of all analytical methods incl. method validation and sample chromatograms for: blank, LOQ standard, spike at LOQ and sample generated during the course of the study.
- complete description of calculation and statistical procedures
- a description of all circumstances which may have affected the quality or integrity of the data
- location of archival of the final report, raw data and test item sample
- a quality assurance compliance statement including dates of inspections
- Good Laboratory Practice Compliance Statement
- copy of DR.U.NOACK-LABORATORIEN GLP Certificate

12 Archiving

RAW DATA

Records to be maintained and provided in the raw data by the Study Director include, but are not limited to, the following:

- original of the study plan, amendments, and deviations
- test item shipping and receiving records, including the Material Safety Data Sheet (MSDS) and Certificate of Analysis (COA), which will be provided by the sponsor
- a list of equipment used in the study
- telephone conversation records and all written correspondence with the sponsor
- SOP deviations, if any, and their impact on the study, along with notification to the Study Director
- documentation of the date of receipt, source, identification, and pre-test maintenance conditions of the test system
- test item solution preparation records, including test item solution calculations and dilution records
- environmental data collected during the test conduct (e.g. temperature, relative humidity, etc.)
- all original data collection sheets

ARCHIVING

The following will be retained in the in-house archive of the test facility for the period as specified in the operative national GLP regulations (15 years):

- all raw data (see above)
- study plan
- final report
- all records performed by the quality assurance programme including master schedules
- samples of test and reference items

Additionally microfilms will be retained in a safe-deposit by Volksbank Sarstedt, D-31157 Sarstedt.

DISPOSAL

After expiry the tests items, standards and analytes will be disposed as difficult waste on garbage dump of Heinde. Disposal prior to expiry will be done in arrangement with the sponsor.

13 Literature

1. OECD 307 Guideline for Testing of Chemicals, Aerobic and Anaerobic Transformation in Soil, Adopted 24th April 2002
2. OECD Principles of Good Laboratory Practice published in ENV/MC/CHEM(98)17, OECD, Paris, France.
3. Overview of the EPA Quality System for Environmental Data and Technology published in EPA/240/R-02/003, November 2002
4. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5 published in EPA/240/R-02/009, December 2002
5. EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 published in EPA/240/B-01/003, March 2001
6. EPA Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8 published in EPA/240/R-02/004, November 2002

14 Study Plan Approvals

14.1 DR.U.NOACK-LABORATORIEN

We, the signatories, declare that this study will be carried out in compliance with the present OECD, EC and German principles of Good Laboratory Practice.

All information, including that provided by sponsor and relevant to this report will be treated confidentially. This includes all data recorded during the course of this study. All reports and results relevant to this study remain the property of the sponsor.
They will not be given to third parties by DR.U.NOACK-LABORATORIEN without the express written consent of sponsor.

Sarstedt,

Date — Study Director
(Silke Fiebig)

Sarstedt,

Date — Scientist for LC-MS/MS analysis
(Dirk Schulze)

Sarstedt,

Date — Scientist for GC-MS analysis
(Thomas Geffke)

Sarstedt,

Date — Head of Quality Assurance Unit
(Gudrun Möhrmann-Kalabokidis)

Sarstedt,

Date — Head of Testing Facility
(Dr. Udo Noack)

14.2 Sponsor

The sponsor's signature signifies consent to the planned test procedure.

14.2.1 ASAHI GLASS CO., LTD.

Place Date ASAHI GLASS CO., LTD.

14.2.2 CLARIANT GMBH

Place Date CLARIANT GMBH

14.2.3 DAIKIN INDUSTRIES, LTD.

Place Date DAIKIN INDUSTRIES, LTD.

14.2.4 DUPONT CHEMICAL SOLUTIONS ENTERPRISE

Place Date DUPONT CHEMICAL SOLUTIONS ENTERPRISE